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All drawings by Milton Franklin Sonday, Jr. in collaboration with the authors.

PRINCIPLES OF TEXTILE CONSERVATION SCIENCE NO. IV

THE CONSERVATION OF HISTORIC TEXTILE COLORANTS

JAMES W. RICE

In Textile Conservation, Why Should We Know Much About The Coloring Substances Employed?

One of the more important tasks we have to do in handling a textile is that of saving and if possible brightening the colors. Usually this is best done by cleaning but in order to do so safely we must know what constitutes a color, how it is applied to the goods, what chemicals affect it and how, and what other factors cause it to fade or change.

Some of the color losses that cleaning cannot help are fading due to light and oxidation damage which we usually term "ageing." Other colorant changes are from surface damage and luster reduction because of mechanical wear. Color restorations from cleaning may be expected by removal of surface soil and stains. Careful treatment of color change effects with chemicals may also help. This particular discussion is designed to provide a background and reasons for practical restoration procedures.

How Are The Main Textile Colorants Classified?

For our purposes, the textile colorants may be classified into four general groups. These are not hard and fast categories, but for organization of our knowledge we list them as follows:

(a) *Stains.* We like to think of stains as non-permanent soluble colorants. They may be formed intentionally or accidentally and usually respond to intelligent removal techniques.

(b) *Dyes.* In our system, dyes are color-forming substances intentionally applied to the fiber by special treatments to produce, if possible, a permanent concentrated color. The color chemicals, whether natural or synthetic, are often called "dyestuffs." These form an exact hue which may, however, be modified or influenced by other chemicals or other dyes of a different hue. The term hue is explained later. An example of chemical influence may be seen when acid is dropped on Congo-red, a cotton dye, and a blue-black spot develops. On the other hand, a blue dye and a yellow dye of the same dyeing class when mixed will form a green hue.

Most dyes are capable of being dissolved or distributed in a liquid in finely divided micelle form for application. Dyes in the right place and condition are desirable but often in cleaning they dissolve or solubilize and spread to some area of different hue to form a stain. This is called "dye bleeding." A knowledge of the classification and properties of the different dyes is very helpful in stopping and controlling "bleeding" during cleaning. When a dye "bleeds," it is said to be "fugitive" or is called a "fugitive dye" to the particular solvent that dissolves it from the fabric. Some writers also term colors that fade because of photochemical effects as "fugitive to light."

(c) *Lakes and Pigments.* Color lakes are insoluble colored substances formed by chemical reactions. This phenomenon may be accomplished as a dyeing operation by impregnating the fabric with one soluble ingredient followed by another soluble substance which reacts with the first to form an insoluble precipitate on the surface or within the pores of the fiber. Lakes can also be made in reaction vessels and not in contact with yarns or fabrics. In this case the product is pulverized and mixed with an adhesive and applied as a pigment by painting or printing on the fabric. The pulverized lake is then called a pigment. Indigo is an example of a dyestuff used in the past in preparing such a pigment.

Many of the pigments occurring in old or primitive textiles are finely divided minerals. Natural iron compounds occur as brown, yellow and red clays and ores. Copper minerals are usually green or blue. A few salts or minerals of other metals have been mentioned occasionally in conservation literature.

The chief requirements for textile printing or painting pigments are that they must be able to be pulverized to very fine soft granules which must be compatible with adhesives so that they will not rub off or flake away from fabric. Some sharp fracturing minerals, although highly colored, lack spreading or covering power and tend to cut and abrade the textile. These are unsuitable for use on fabrics. Ground, colored glasses fail for the same reasons. It is unlikely that we will encounter any of these but if we do, look out for weakened yarns.

Finely divided carbon black, from charcoal, is another common example which produces a soft non-metallic black or gray pigment that is insoluble and lightfast but not really wash fast. For that, it must be put on with an adhesive. Carbon black may be seen in very old specimens which are hard to clean satisfactorily.

From the conservation point of view, the adhesive is the weak point of pigmented textile coloring matter because when it ages, it either becomes hard and chips off or pulverizes and dusts away or the adhesive quality fails and the pigment is easily removed by washing.

(d) *Soils.* The effect of soils on textile colors is important in connection with conservation. Soils are unintended colorants which are a result of accidental smears by contact, atmospheric deposits, wetting stains or oxidation and photo-chemical changes in the fibers. Most soils are black, brown, gray or tan and have a dulling effect on the base colors. The source of the soil and the conditions under which it deposits govern the tenacity with which it sticks. This will be explained in a later paper on cleaning and detergents.

What Are Some Of The Terms Used In Describing And Defining Color Qualities?

White sunlight if analyzed by a prism or as in a rainbow is made up of a number of *hues,* such as red, orange, yellow, green, blue, and violet. Red, yellow and blue constitute the *primary hues.* Mixing any two pigments of the primary hues gives *secondary hues.* Thus orange is a mixture of red and yellow; green of yellow and blue, and violet of blue and red. Any mixture of all three is called a *tertiary hue.* A very great number of different hues, each with its own radiant energy wave length, is possible. When all of these are combined, we have a broad wave length band of white light called the *spectrum.*

A primary or secondary hue at its maximum purity becomes a *brilliant hue.* A mixture of the three primary hues in equal parts produces a gray. The measurement or descriptive term for brilliancy or purity of a hue away from the neutral is called its *chroma.*

Difference in grays from black to white or in the *shades* or *tints* of the hues are known as color values. Note that grays may be formed of mixtures of three primary hues, a mixture of a primary and an opposing secondary or complementary hues, or of black and white.

The diagrams in Figures 1 and 2 may illustrate these terms.

Because the color estimate depends upon three parameters, chroma, value and hue, a precise evaluation of any particular color in numerical terms is difficult and color judging by eye is still probably better than by instruments. This becomes evident when we try to match colors exactly in redyeing or tinting a faded area of a textile or when choosing yarns for repairing or darning a damaged section of the fabric. For that reason, all three of these color characteristics must be considered in colorant restoration problems, where training and experience in visual color differentiation is of great value.

What Are Some Of The Molecular Structural Peculiarities of Dyestuff Molecules?

Most dyestuff molecules are comparatively large in size. As a rule, large molecules are relatively insoluble in cold water but may dissolve more readily in hot water. Most dyes behave in this manner and for this reason the dye bath is heated, sometimes to boiling. On cooling, these large molecules agglomerate to form precipitates of gels or solid particles which will then deposit from solution.

Figure 1

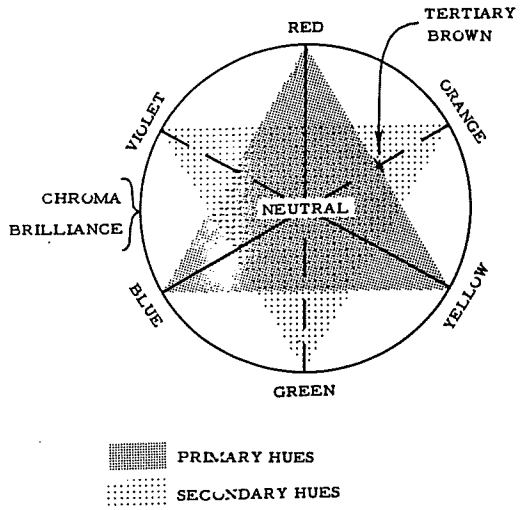
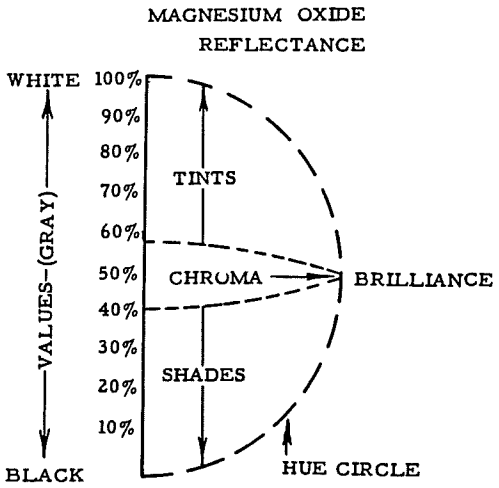


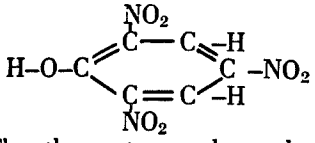
Figure 2



All dyestuff molecules contain *chromophores.* These are special groupings of chemical elements which occur in a conjugated or definitely repeating order in the molecular structure. The number of chromophores in the molecule determine its color strength. There are more than a dozen of these chromophores or special atomic groupings such as: $-C=C-$, $=C=C=$, $-C\equiv C-$, $-C=O$, $-C=N-$, $-N=N-$, $-N\begin{matrix}O\\ \diagup\!\!\diagdown\\ O\end{matrix}$, $-C=S$ and others. Whenever any of these rather unstable double bond connections between atoms in the chomophore group are changed or affected by some outside influence, the hue of the dyestuff changes. This accounts for fading because the bonds may be broken by ultra violet radiant energy in the shorter wave lengths, by oxidation or reduction or in some cases temporarily parted by the influence of acids or alkalies (pH).

Unless the color molecule can be fixed to the fiber it will merely form a non-permanent stain which will wash out easily. Dye chemists overcome this by building into the molecule certain other chemical groups which are called *auxochromes.* These are reactive groups or radicals which promote solubility. The auxochromes are also supposed to intensify the color. Important auxochrome groups or radicals are $-NH_2$, $-OH$, $-OCH_3$, $-NHCH_3$, $-O-SO_2$ and others.

To illustrate the terms chromophores and auxochromes we offer picric acid, or trinitrophenol. It is one of the earliest synthetic dyes discovered. It is a brilliant yellow which has great affinity for proteins and may be encountered in fabrics dating from the last decade of the 18th century until after the middle of the 19th century. It is no longer widely used because it is an explosive also and materials dyed with it may be flammable. Its structural formula as drawn in a plane looks like this:



Note: The three strong chomophores $-NO_2$ located at regular intervals around the structure to influence another conjugated chromophoric group $-C=C-C=C-C=C-$ to which the auxochrome HO– is attached.

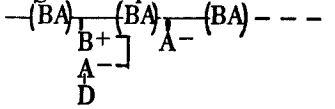
Such chromophoric groups are doubly important because the unstable chemical bond configurations not only reflect certain individual wave lengths or bands of white light but will absorb other wave lengths in the infra red spectrum. This permits identification by modern infra red spectrophotometry.

From The Conservation Point Of View, How Are Dyes Best Classified?

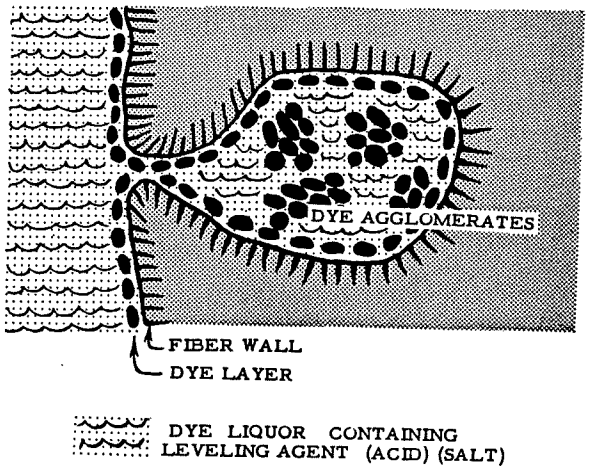
Although many writers classify the dyes of history into rough source categories such as vegetable, animal and mineral, and modern dyestuff chemists use a system of assigning recent discoveries into chemical family groups, the textile conseravtion worker will find another, based on the behavior of the dye substance in dyeing of most value in solving cleaning and restoration problems. This is used by professional cleaners who, for the same reasons, use the following system to advantage because cleaning, in general, is an opposing effect to dyeing. There are at least twelve divisions in this system but we are at this time interested in five only: Acid Dyes; Basic Dyes; Direct Dyes; Mordant Dyes, and Vat Dyes. These are presented in the following discussions of their chemical and physical behaviors when being applied and during cleaning. A final condensed table of these characteristics is also provided as a summation to this paper.

What Are Acid Dyes?

Acid dyes are so named because they require acid solutions to control their union with the fiber. At the moment of dyeing they actually become acids which unite with the basic sites on a protein fiber molecule to form an insoluble complex. For this reason they will combine directly with protein fibers but not cellulosics. If we represent the protein fiber by the diagram in our previous discussion, No. III of this series of papers, *Classification of Fibers Found in Ancient Textiles,* as $-(BA)-(BA)-(BA)-$ and an acid dye at the reaction moment as $A^-—(D)$, the combination might be represented thus:



The acid solutions used for control may be dilute mixtures of sulfuric, formic, acetic or other soluble acids. In modern dyeing practice these added acids are called *leveling agents.* They not only control the rate that the acid dyes are absorbed but also their solubility and one way of increasing the amount of dye deposited in the pores of the fiber may be imagined in the following sketch of the inner pore of a fiber:



In this case the stronger acids cause the molecules of dye to collect into micelles or relatively large particles in gel form which can no longer escape from the pore. In cleaning work, the acid is called a setting agent because it is used to prevent the excess dye micelles from dispersing and moving out from the pores. *Alkalies*, which are strong *bases* are the opposites of the acids and will neutralize them. Thus alkalies will cause the dyes to dissolve. This is a *bleeding* agent.

Of the the several acids named, the only one that was probably used in ancient or primitive dyeing practices was acetic acid, which is found in vinegar, and is a product of fermentation of glucose, a widely occurring sugar. Since very many of the natural occurring dyestuffs are glucosides or a combination of glucose and acid colorants, a dye job might be produced by fermenting the glucoside prior to or during the actual dyeing operation to produce the acidic conditions necessary. Otherwise the freshly prepared color substance is not wash fast.

Coagulation of the dye particles or gels is also opposed by detergents and hot water. The detergent disperses the micelles and prevents agglomeration. It also lubricates the pore entrances. In dyeing practice, it is called a *dyeing aid.*

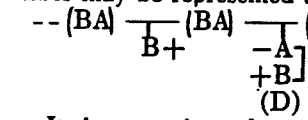
The hot water or temperature effect swells the fiber and opens the pores permitting the entrance of the dyes. It also affects the dispersion of the dyes. On dropping the temperature, the dye particles coagulate and the pores close in to hold them. Some dyes are soluble at a relatively low temperature, while others will not dissolve until the water boils. These tendencies are employed often in dyeing with mixed colors. For example, in dyeing a fabric a golden orange, a mixture of red and yellow dyes is dissolved in boiling water and the yarn entered. One of the colors is deposited at once, as for example, the red. When the temperature is dropped, perhaps 30 degrees, the second color is deposited, producing the desired hue. This accounts for some cases of color change in cleaning when one of the components bleeds and escapes before others in relatively cool water.

There are numerous examples of natural dye substances, used from antiquity on as reported in the history of dyes, which indicate that most of the better natural colorants were fundamentally acid dyestuffs. Of a goodly number of the natural dyestuffs, the red hues, almost all of the oranges and yellows, a majority of the browns, occasional greens, one or two of the blues, and rare purples may be presumed to be of this class. We offer these in this order, in hopes that it may be a clue to future infra red, visible white, and ultra violet radiant energy analysis interpretation techniques.

Since acid type dyestuffs usually bleed to alkali solutions such as ammonia, this is used as a single recognition test. If acetic acid stops the bleeding, the test is confirmed. This tells us that alkalies should be avoided in cleaning and that acid solutions are useful in counteracting them.

What Are Basic Dyes?

The basic dyes are substances containing an amine group which will attach itself to an acid site on the protein molecule in the same way that acid dyes unite with the basic protein sites. Thus if we picture a basic dye in this symbol D $-B^+$ the dyed fiber may be represented thus:



It is a curious fact that basic dyes may be accepted by the protein fiber after it has been dyed by an acid dye. This is called topping.

In our studies of dyeing history we have encountered only a very few basic dyes that we recognize as such among those used in ancient and medieval times. They are more apt to be found on protein fibers from about 100 years ago when the modern dye manufacturing began. They are not very light fast and older examples will probably be badly faded. The colors are likely to be quite brilliant if they have not been exposed to light.

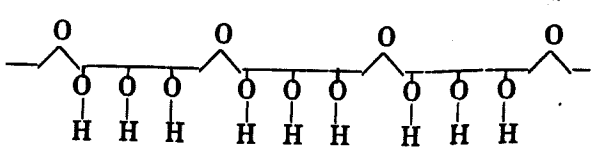
Cotton does not accept basic dyes unless it is mordanted. This will be discussed later. Jute fiber, straw and wood as in baskets and other mat fabrics contain lignocellulose or lignins which are slightly acidic. Therefore, basic dyes will combine directly with such fibers. For that reason, the conservator may encounter them in semi-primitive colored museum specimens.

Dyeing techniques on fabrics are very similar to those used for acid dyes except that leveling agents, if necessary, are either alkalies such as ammonia or sometimes neutral salts such as sodium sulfate.

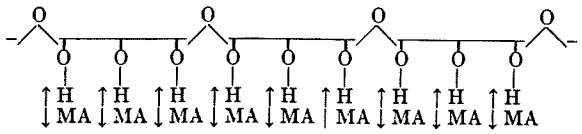
Basic dyes may be dissolved by acids whereas alkalies tend to set them. This behavior is used as a simple identification test. If bleeding is detected on a white blotter when a spot is moistened with acetic acid for example and if this ceases on neutralization by a drop of ammonia, we know that acid setting agents (sours) should be avoided in cleaning routines.

What Are The Direct Cotton Dyes And How Do They Work?

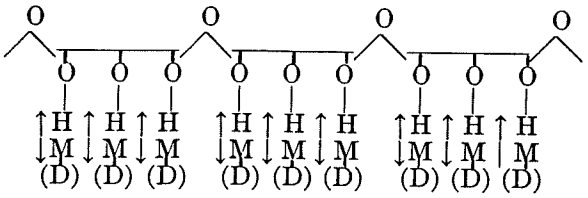
Cotton or cellulose, as has been explained in previous papers dealing with the fibers, does not react directly with acids or bases. For that reason, it has long been considered as hard to dye. It will accept special types of molecular combinations, however, in a surface attraction effect which the following diagrams may help to explain. The long cellulose chain is characterized by many exposed hydroxyl (–OH) groups and occasional oxygen links which oversimplify thus:



These –OH groups have an attraction for molecules containing similar groups as, for instance, water H–O–H, alkalies M–OH where M stands for a metal, and M–A where M represents a metal combined with an acid to form a salt. These substances are attracted to the surface thus:



This junction is much weaker than that of acids or bases in the case of proteins. The direct cotton dyes resemble the above metallic salts in that they are also salts of acid dyes and metal hydroxides. Hence the surface attraction affinity is much the same and our mental picture, where M–(D) represents a dye-metal salt, is:



This attraction <——> is actually weak and the dyeings depend mostly on the use of the coagulating and leveling agents to cause the dye particles to deposit within the pores, as is shown in the sketch of the inner pore of a fiber (page 57) to confer wash fastness which, however, is not as good as some of the dyeings on wool.

The leveling and coagulating agent in this case is either sodium chloride (common table salt) or sodium sulfate (Glauber's salts) both of which are neutral.

Dyeings are in hot water baths but careful attention must be paid to the exact temperatures used for each individual dyestuff.

Saffron, a yellow dye of antiquity, is said to be an example of this direct cotton class and is noted for its fugitive nature both to washing and light. Hence it was never considered a really satisfactory dye. Modern direct cotton dyes are very much better, however, and as a rule have satisfactory light fastness.

A rough practical recognition test is made as follows: Saturate a small area of fabric with a hot soap or detergent solution and after a few seconds apply a white blotter under pressure. If color shows, it may be a direct cotton dye. If upon wetting the test area with a 5 to 10% brine solution, the blotter no longer picks up dye, the test is probably confirmed.

In cleaning, one should avoid hot alkaline suds solutions and if bleeding occurs, transfer the work immediately to a cool brine rinse bath.

What Are The Mordanted Dyes and How Do The Mordants Function?

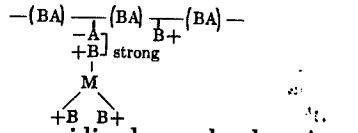
The acid, basic and direct dyes produce a great range of beautiful colors but in many cases the dyeings are not as fast nor as permanent as we may desire. One way of increasing the wash fastness is by mordanting. This was discovered very early in history.

A *mordant* is a dyeing aid which forms an insoluble color lake in and on the fiber. There are two classes of mordants, the basic and the acidic types. The best of the basic types are easily hydrolized salts of tri- or tetra-valent metals such as iron, aluminum, chromium and tin. However, copper, zinc, lead and mercury are a few di-valent metals that are occasionally encountered in mordants. In ancient and medieval dyeings, aluminum in the form of alum, a natural occurring mineral, iron in certain spring waters and also from natural mineral sources, tin components from chemical attack on pewter and block tin vessels and copper from similar sources and from mine waters may have been used.

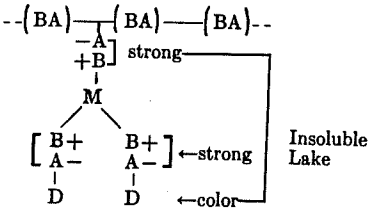
The acid mordants are either vegetable substances containing tannin similar to gallic acid from oak galls and tree barks or they may be vegetable oils. The latter are uncommon. The tannins unite with proteins such as raw skin or hides to form insoluble precipitates in leathers and since they have properties of dyes and cause dulling, or reduce hue chromas, they are used principally for that purpose rather than for mordanting. In modern practice, they are employed extensively for mordanting cotton to receive basic dyes.

In doing its job, the mordant reacts first with the fiber surface forming a sort of chemical bridge between the fiber and the dye which when it comes in contact deposits out in an insoluble form. Assuming the mordant to be either aluminum or iron, its molecule may be represented as

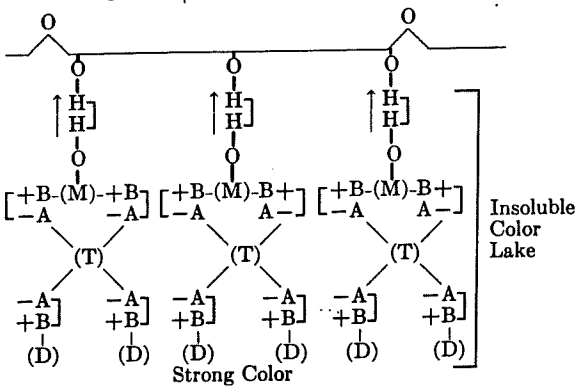
When this comes in contact with the protein, it joins at the acid A sites thus:



When an acidic dye molecule $^{-}A-D$ comes in contact with the mordant basic sites, a firm strong junction occurs and since these tri-valent metals form insoluble salts with very weak acids, a dyeing of great permanency results. The bridge may be imagined thus:



The alum (or other soluble metallic salt) solution is worked into the fiber cold and then brought to boiling. This converts the alum to Al $(OH)_3$ aluminum hydroxide, a sticky, jelly-like substance which, if it does not react as shown above, plates out on the pores and outer surfaces. This is what happens in the case of cotton. Thus cotton will receive acid dyestuffs if mordanted with alum and then is boiled or steamed. Should it be desirable to dye the cotton with a basic dye, we mordant first with alum and follow it later with tannins. These will then accept the basic dyes. This is then our picture:



Barberry, probably one of the few natural ancient basic dyes, was mordanted on cotton using tin salts and tannin in this manner.

Variations in hues may be achieved with the same dye by using different mordants. For example, madder produces an excellent red when mordanted with alum and a deep black when mordanted with iron. Some similar effect may have accounted for Heroditus' tale of the sheep that came out in different colors when dipped in the same dye bath.

There are no simple positive tests for the mordanted colors except if tests for acid, basic, or direct cotton types are negative, we may assume that we are dealing with a "lake" of some kind. If more definite information is demanded, a complete chemical or even an X-ray analysis may be necessary.

Cleaning presents no particular problem because of the insoluble nature of the mordant lakes. These may be affected by continued exposure to hot concentrated alkalies or acids. Since such treatments are unlikely because of their damaging effect on most fibers, it is improbable that any damaging effects will happen.

What Are The Vat Dyes And How Are They Applied?

Vat dyes are among the most satisfactory colorants found in old textiles. They are fast to washing and their colors improve with age. Indigo and woad, both vegetable substances which are fundamentally the same chemical compound and Tyrian purple, an animal product very closely related chemically to indigo, are classic examples of this group.

The name comes from large vats originally used to prepare and ferment the roots of the indigo plant or stalks of woad before the color could be formed in the fabric. Upon fermentation, the blue indigo color changes to a pale yellow water soluble state. This is called the *leuco* or reduced form and is soluble at that time in alkaline solutions. The fabrics or yarns are then dipped in the leuco solution and exposed to the air, whereupon oxygen combines with the reduced dye substance to form a blue colored insoluble deposit which cannot be dissolved by ordinary washing and is not affected by light or oxidation. Depth of color ranging from green through to almost black may be achieved by repeated dippings.

Indigo was very widely used in ancient times and has even been found in old Peruvian textiles. It is somewhat more concentrated in dye substance than the product obtained from woad which was long used in Northern Europe and has been traced even to early Egypt.

Indigo dyes both cotton and wool but is more apt to be found on cotton because of the damaging effect of alkalies, used to make it soluble, on wool. Early dyeing recipes mention the use of stale urine, which is alkaline because of ammonia formation, as an ingredient in dyeing with woad. This was for dissolving the leuco compound. Bran was also used to ferment and produce the reducing action on the woad. This particular recipe was probably designed for dyeing wool because neither of these ingredients will damage it appreciably.

Tyrian purple is produced by several species of shell fish inhabiting various sea shores scattered over the world. Its chemical name is 6,6′ dibromindigo. In ancient times it was very expensive and was used principally for dyeing garments for royalty. It was one of the principal exports of the city of Tyre in the eastern Mediterranean and it is associated with that part of the world although ancient Peruvian and Central American textiles have been found that were colored with this chemical compound.

As produced directly by the shellfish, the dye is in the *leuco* form and if applied to the yarn immediately, the color begins to develop. The leuco color is yellow but in successive stages it turns orange, scarlet, crimson, purple, and eventually a deep violet. This change may be very slow but it can be brought about quickly by drastic oxidation such as bleaching. It dropped out of use in Europe during the Middle Ages. Whether it was still available to the Near Eastern people after the Crusades is questionable. About the turn of the present century, it was synthesized in the laboratory. The colors of the 6,6′ dibromindigo proved to be dull and not satisfactory but a close relative, 5,5′ dibromindigo was found to be a much better product. This is probably more available of the two today.

A practical test for one of these vat dyes is as follows: Select some inconspicuous part of the textile specimen for the experiment. Wet a tiny area of the unknown color with a drop of neutral

detergent. Add another drop of acetic acid. Follow with a drop of commercial dye stripper (sodium hydrosulfite 2%). This is a powerful reducing agent. Observe this for a few minutes and if the color fades to a pale green, orange or yellow, it may be a vat dye. If another drop of 1 to 3 % hydrogen peroxide causes the test spot to return to its original or even deeper hue, the test is confirmed.

The results of vat dyeings are lakes which are not affected by washing. However, the above recognition test indicates a conservation problem. Rust stains, ink spots and areas of overoxidation often appear on vat dyed rug colors as black spots. Treatment with rust removers reverses the color but it may be very difficult to restore it to the original hue. Sometimes pet accidents may bring out the leuco color. In this case, reoxidation by bleaches is a very difficult undertaking. If left alone and given time enough, the colors may return to the original hue.

What Causes Indicator Type Color Changes In Dyes And How Can They Be Overcome?

In addition to the oxidation-reduction color change in the vat dyes as described previously, the conservator may occasionally see a change in hue because of changes in the pH or acidity or alkalinity of the cleaning solutions. This phenomenon is occasioned by highly unstable chromophores which are affected by hydrogen ions. As an example, turmeric, a brilliant yellow vegetable dye of antiquity is normally slightly acid and yellow. In presence of ammonia, an alkali, it turns a dark reddish brown. With acid again, the yellow returns. Many other vegetable acid dyes, stains and mordanted dyeings react in the same way. Such unstable colorants are called *indicators* because they are valuable chemical tools for indicating when an analysis, for example, has been completed. Litmus, which is familiar to most elementary science students, is one of these. Obviously, the cleaner in such a case should change the acidity of the solution.

TABULATION OF PROPERTIES OF DYE CLASSES

CLASS	EXAMPLES	SUGGESTED TESTS	CLEANING PRECAUTIONS	SETTING AGENTS (suggestions)
Acid	Turmeric Annatto Brazil wood	Try detergent with a little ammonia for bleeding evidence to pressure with blotter.	Avoid hot alkaline soaps. Watch prolonged soaking in neutral solutions.	Acetic or other mild acid, especially when rinsing.
Basic	Archil (Litmus) (Orseille) Persian Berry	Try a neutral detergent first, followed by acetic acid for evidence of bleeding to pressure on a blotter.	May withstand very short exposure to mild alkalies. Avoid mild or strong acids. Be wary of even moderate exposure to neutral detergents.	Very weak ammonia solutions, especially if quite cold.
Direct Cotton	Lo Kao (Chinese green) Saffron Safflower	Try hot water or soap solutions for evidence of bleeding on a blotter	Avoid long periods of soaking in hot soap suds or alkaline detergents.	Salt brine followed by cold water.
Mordant	Cochineal Lac Logwood Weld Kermes Madder (Turkey Red) Safflower Annatto	Try in turn suggested tests for acid, basic and direct groups. If negative, dye is probably mordant. Do not be fooled by color changes. ..... Try drycleaning solvent on "Turkey Red."	No special warnings necessary except in the case of "Turkey Red" which may bleed in drycleaning.	Cold water alum.
Vat	Indigo Woad Tyrian Purple	Try reducing agent followed by mild bleach to set.	Avoid reducing agents in connection with alkalies.	Very dilute mild bleaches.